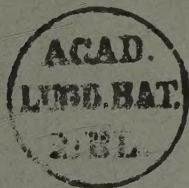

THE BURNING, OVERHEATING, AND RESTORING OF NICKEL STEEL.

G. B. WATERHOUSE.

Submitted in partial fulfillment of the requirements for the degree of
Doctor of Philosophy in the Faculty of Pure Science, Columbia Univer-
sity, New York City.

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BY G. B. WATERHOUSE.

The following paper is based on an investigation conducted in the metallurgical laboratory of the School of Mines of Columbia University in the City of New York.

The author has much pleasure in acknowledging the ready counsel and aid of Professor Henry M. Howe, who planned the work of investigation, and under whose immediate direction it has been carried out; of Professor Bradley Stoughton, and the other members of his staff. Thanks are also due to Professor I. H. Woolson, of the Mechanical Engineering Department, Columbia University, for allowing the mechanical tests to be made in his laboratory.

OBJECT OF THE INVESTIGATION.

The object of the following research is to ascertain the temperatures required to injure nickel steel by overheating, and to remove the injury thus caused, studying the changes by means of mechanical and microscopical tests.

Nothing is known of this subject by direct observation, and we have only the analogy of common steels.

The literature on the Burning, Overheating, and Restoring of Steel has been recently reviewed by the author.* The following definitions were proposed:

Burnt Metal.—Coarsely crystalline and exceedingly brittle iron or steel, in consequence of excessive heating; often with some inter-crystalline layers of oxide of iron. It cannot be effectively restored by heat treatment or mechanical work.

Overheated Metal.—Coarsely crystalline and brittle iron or steel, in consequence of excessive heating; with no inter-crystalline

* G. B. Waterhouse. The Burning, Overheating and Restoring of Steel. *Engineering and Mining Journal*, Vol. 81, 1906, pp. 386, 414.

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spaces. It can be restored by heat treatment or mechanical work.

Restoring.—The operation of removing the injurious effects of excessive heating, and attempting to restore the initial or superior properties to the material.

The results arrived at were that steel becomes burnt on heating to a temperature above the line Aa in Fig. 1.

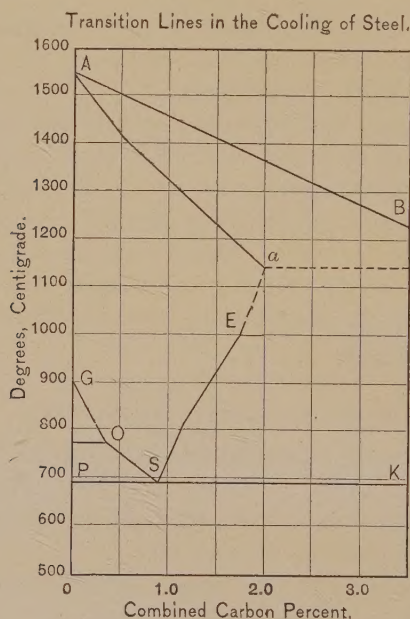


FIG. 1.

Such steel cannot be effectively restored. When heated to temperatures below Aa, steel may become overheated; from which condition it may be restored by heating to a temperature slightly above G O S E where solid solution is complete.

EXPERIMENTAL METHODS AND LINE OF RESEARCH OF THIS PAPER.

A series of seven crucible steels were taken in which the nickel was constant, the carbon rising from .41 per cent. to 1.52 per cent. and the other elements as low as possible.

The mode of preparation of these steels, and their properties under several conditions have been previously described by the author.* The analyses of the 1 inch diameter rolled bars are given in Table I.

TABLE I.

Mark of Steel.	K.	L.	M.	N.	O.	Q.	R.
Total Carbon, per cent.	.41	.51	.63	.79	.97	1.24	1.52
Nickel, per cent.	3.79	3.79	3.76	3.81	3.75	3.81	3.82
Silicon, per cent.	.102	.103	.133	.129	.095	.117	.144
Manganese, per cent.	.086	.054	.050	.051	.058	.057	.054
Sulphur, per cent.	.013	.014	.014	.014	.014	.014	.015
Phosphorus, per cent.	.007	.008	.008	.008	.008	.008	.007
Aluminium, per cent.	.010	.010	.009	.010	.011	.009	.010

The critical points are given in Table II.

TABLE II.

Mark of Steel.	K.	L.	M.	N.	O.	Q.	R.
Heating. Ac. 2.3 degrees Centigrade	744	734					
Heating. Ac. 1 degree Centigrade	704-714	699-714	702-717	703-718	701-716	688-698	683-693
Cooling. Ar. 3.2 degrees Centigrade	653-638	640					
Cooling. Ar. 1 degree Centigrade	608-598	615-605	610-600	612-602	612-602	620-610	621-611

As compared with carbon steels the transformation points, Ar 3, Ar 2, and Ar 1, have been lowered about 20° C. for every 1 per cent. of nickel.

To determine the overheating temperatures, bundles of bars, 10 inches long, consisting of one bar of each steel, were heated to 1000° C., 1100° C., 1200° C., 1300° C., 1400° C., and 1460° C. The heating was carried out in a gas fired crucible furnace, using the arrangement shown in Fig. 2. The time required to heat to the first four temperatures was 1 hr. 10 min., 1 hr. 45 min., 2 hr. 35 min., and 2 hr. 20 min., respectively. The bars, in all cases, were maintained for an hour at the desired heat, and cooled slowly in place. The mechanical tests of these bars were then made, and a section cut off for microscopical examination.

* G. B. Waterhouse. The Influence of Nickel and Carbon on Iron. *Journal Iron and Steel Institute*, No. II, 1905, p. 376.

The overheating temperatures having been ascertained, bars of each steel were heated to this temperature under similar conditions. Bundles were then made up as before of one bar from each steel, and these bundles reheated to 700°C . and 775°C ., held there for one-half hour and cooled in the furnace. The reheating

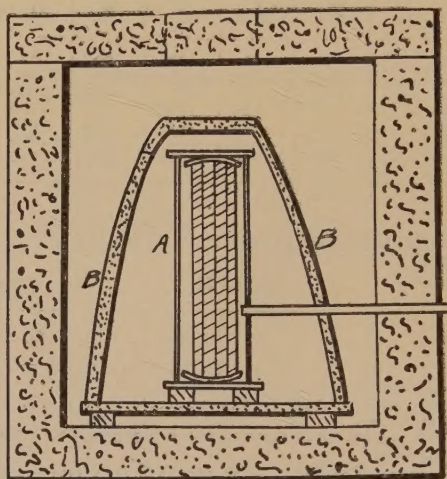


FIG. 2.—A, Mild Steel Pipe.
B-B, Graphite Crucible.

was carried out in a special gas-fired muffle furnace, designed by Professor Howe, of which a description is given in a paper by him, published simultaneously with the present one.

The results may be conveniently classified under the heads chemical, mechanical, and microscopical.

CHEMICAL.

The analyses of the steels have been given in Table I. The separation of temper graphite in the upper members of the series is very interesting. The results are given in Table III, including those published in the preceding work.*

* G. B. Waterhouse. The Influence of Nickel and Carbon on Iron. *Journal Iron and Steel Institute*, No. II, 1905, p. 376.

TABLE III.

Mark	Condition of Carbon.	Ingot.	Billet.	Rolled.	Treat-ment A. 1000° C., cool in a r.	Treat-ment B. 875° C., cool slowly.	Heated to 1000°.	Heated to 1100°.	Heated to 1200°.	Heat'd to 1300°.
Q.	C. C.	1.24	1.24	1.24	1.24	1.24	.73	1.16	1.24	1.24
	Gr. C.						.51	.08		
R.	C. C.	1.52	1.05	1.48	1.21	.50	.23	.29	.83	1.52
	Gr. C.		.47	.04	.31	1.02	1.29	1.23	.60	

Heated to 1000°. Reheated 700°.	Heated to 1200°. Reheated 775°.
1.24	1.24
....	
.54	.53
.98	.99

Treatment A consisted in heating the bars to 1000° C., which took 1 hour 25 minutes, holding them there for 25 minutes and cooling them in air. In treatment B, the bars were packed in a wrought iron tube, placed in an annealing furnace at 500° C., and brought to 870°-880° in 5½ hours. They were held there for 3 hours and then slowly cooled. The times of the other heatings have been given in the experimental methods adopted.

In the steels subjected to treatment B, the cementite was found* to have a composition represented by the formula $\text{Fe}(\text{Ni})_3\text{C}$. Its analysis was:

Carbon, per cent.	6.25
Nickel, per cent.	1.86
Iron, per cent.	91.71

MECHANICAL.

The bars were turned down to a diameter of 0.564 inches, giving an area of 0.25 square inches, for a length of 2 inches.

The results are given in Tables IV to X, and also shown in Diagrams I and II.

* G. B. Waterhouse. The Influence of Nickel and Carbon on Iron. *Journal Iron and Steel Institute*, No. II, 1905, p. 376.

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TABLE IV.—Steel K. C. C. .41 per cent.; Ni 3.79 per cent.

Treatment.	Elastic Limit.	Ultimate Stress.	Elongation per cent. on 2 in.	Reduction of Area per cent.	Fracture.
	lb. per sq. in.	lb. per sq. in.			
Treatment A	48563	90246	26.0	44.7	Half cup and cone. Gray granular.
Heated 1000°		71800	26.0	48.2	Half cup and cone. Gray granular.
Heated 1100°	44880	71700	25.5	48.6	Half cup and cone. Gray granular.
Heated 1200°		74000	11.0	10.5	Crystalline, level fracture.
Heated 1300°		71320	7.0	4.8	Coarsely crystalline.
1200° reheated 700°	38809	71487	10.5	10.4	Slightly finer than 1200°.
1200° reheated 775°	43515	74989	25.0	32.5	Gray granular, almost complete cup and cone.

TABLE V.—Steel L. C. C. .51 per cent.; Ni 3.79 per cent.

Treatment.	Elastic Limit.	Ultimate Stress.	Elongation per cent. on 2 in.	Reduction of Area per cent.	Fracture.
	lb. per sq. in.	lb. per sq. in.			
Treatment A	50469	99109	21.0	39.05	Half cup and cone. Gray granular.
Heated 1000°		78600	25.0	42.5	Half cup and cone. Gray granular.
Heated 1100°	46600	78800	24.0	42.4	Half cup and cone. Gray granular.
Heated 1200°	47200	84900	11.5	12.1	Crystalline, level fracture.
Heated 1300°		79600	5.0	5.2	Coarsely crystalline.
1200° reheated 700°	46805	81487	15.5	17.9	Crystalline, finer than 1200°.
1200° reheated 775°	47909	80795	22.5	33.1	Gray granular, deeply pitted.

TABLE VI.—Steel M. C. C. .63 per cent.; Ni 3.76 per cent.

Treatment.	Elastic Limit.	Ultimate Stress.	Elongation per cent. on 2 in.	Reduction of Area per cent.	Fracture.
	lb. per sq. in.	lb. per sq. in.			
Treatment A	56041	115421	16.5	26.3	Finely crystalline.
Heated 1000°	47200	80000	20.5	33.1	Gray granular, serrated edges.
Heated 1100°	47200	89400	19.0	32.5	Gray granular, serrated edges.
Heated 1200°		90600	7.0	6.3	Crystalline, level fracture.
Heated 1300°		85200	2.0	1.7	Coarsely crystalline.
1200° reheated 700°		90040	10.0	10.8	Crystalline, level fracture.
1200° reheated 775°	53824	89842	21.0	34.5	Gray granular. Half cup and cone.

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TABLE VII.—Steel N. C. C. .79 per cent.; Ni 3.81 per cent.

Treatment.	Elastic Limit.	Ultimate Stress.	Elongation per cent. on 2 in.	Reduction of Area per cent.	Fracture.
	lb. per sq. in.	lb. per sq. in.			
Treatment A	66850	135194	14.0	21.3	Finely crystalline, serrated edges.
Heated 1000°		108960	15.0	23.5	Finely crystalline.
Heated 1100°	48000	111840	14.0	22.3	Finely crystalline.
Heated 1200°		109600	3.0	3.1	Coarsely crystalline.
Heated 1300°		66800	.5	1.0	Very coarsely crystalline.
1200° reheated 700°		102705	6.0	9.5	Crystalline, finer than 1200°.
1200° reheated 775°	70276	99214	21.0	34.8	Mixture gray granular and fine crystals.

TABLE VIII.—Steel O. C. C. .97 per cent.; Ni 3.75 per cent.

Treatment.	Elastic Limit.	Ultimate Stress.	Elongation per cent. of 2 in.	Reduction of Area per cent.	Fracture.
	lb. per sq. in.	lb. per sq. in.			
Treatment A	80120	156827	7.5	8.4	Fine crystalline, even surface.
Heated 1000°	114121	130336	Broke on shoulder		Fine crystalline, even surface.
Heated 1100°	107313	138112	3.5	4.2	Fine crystalline, even surface.
Heated 1200°		83117	.5	.4	Coarsely crystalline.
Heated 1300°		46648	.0	.3	Very coarsely crystalline.
1200° reheated 700°	66390	114107	5.5	6.3	Coarsely crystalline.
1200° reheated 775°	74437	103476	18.0	19.0	Fine crystalline, and level.

TABLE IX.—Steel Q. C. C. 1.24 per cent.; Ni 3.81 per cent.

Treatment.	Elastic Limit.	Ultimate Stress.	Elongation per cent. of 2 in.	Reduction of Area per cent.	Fracture.
	lb. per sq. in.	lb. per sq. in.			
Treatment A	102030	168607	3.5	3.2	Fine crystalline, very level.
Heated 1000°	70538	97510	15.0	17.9	Mixture gray granular, and fine crystalline, deeply pitted.
Heated 1100°	57982	98183	1.0	.4	Crystalline, level fracture.
Heated 1200°		90729	.5	.0	Crystalline, platy fracture.
Heated 1300°		60600	.0	.0	Crystalline, platy fracture.
1200° reheated 700°	69202	95103	1.5	1.0	Crystalline and level, Similar to 1100°.
1200° reheated 775°	74000	106304	3.5	5.3	Fine crystalline, and level.

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TABLE X.—Steel R. C. C. 1.48 per cent.; Ni 3.82 per cent.

Treatment.	Elastic Limit.	Ultimate Stress.	Elongation per cent. on 2 in.	Reduction of Area per cent.	Fracture.
	lb. per sq. in.	lb. per sq. in.			
Treatment A	65530	145642	10.5	13.9	Crystalline, tinged with gray.
Heated 1000°	35771	63950	23.6	37.6	Deep gray granular. Half cup and cone.
Heated 1100°	40320	66640	25.0	33.5	Deep gray granular. Pitted surface.
Heated 1200°	47157	97894	8.0	5.0	Crystalline, level fracture.
Heated 1300°		35430	1.0	.4	Crystalline, platy fracture.
1200° reheated 700°		89045	17.5	12.2	Deep gray, pitted surface.
1200° reheated 775°	45967	74502	24.0	33.5	Deep gray, half cup and cone pitted surface.

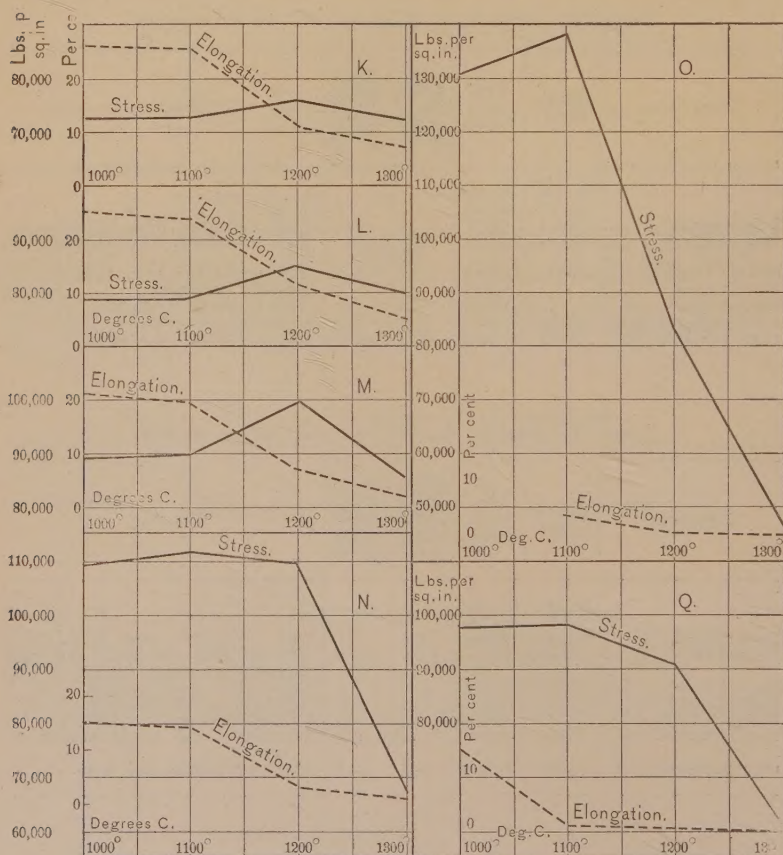


Diagram I. Effect of Overheating on Ultimate Stress and Elongation.

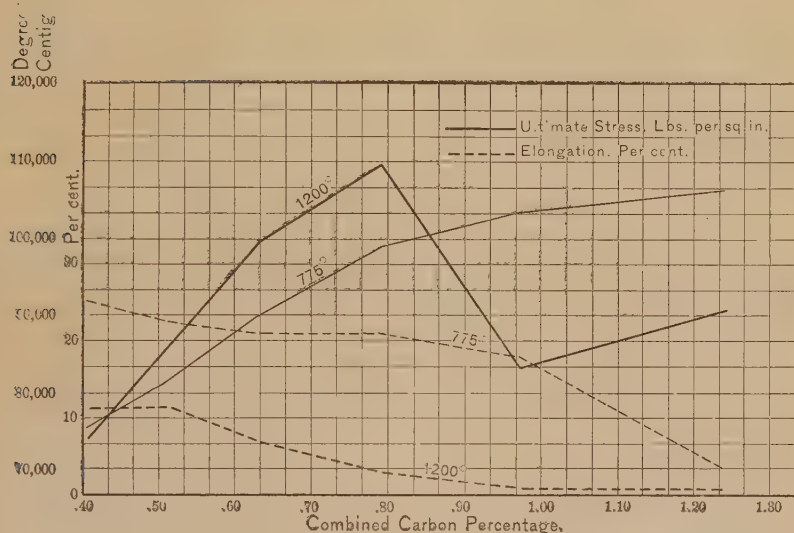


Diagram 11. Comparison of Overheating at 1200°, and Subsequent Reheating to 775°

The growth of the crystals with increasing temperature, and the restoring by subsequent reheating is shown by Photograph I, which represents the fractures of O, the tests of which are given in Table VIII.

MICROSCOPICAL.

Transverse sections were taken of the bars after the various treatments. They were rubbed down on four emery papers of successively finer surface, and finally polished with wet rouge.

The constituents are ferrite, pearlite, cementite, and temper-graphite. Considering, first, the steels submitted to Treatment A; the first one, K, shows ferrite and pearlite in about equal amounts, micrograph 1. With increasing carbon the ferrite diminishes, micrograph 6. In the case of O, it forms the entire field, and Q, with 1.24 per cent. C. C. shows much free cementite, micrograph 10. In R, temper-graphite has separated, shown in micrograph 12, which is from the unetched section; when etched it shows the four constituents present at once, micrograph 13.

The steels heated to 1000° and 1100° are very similar. In the cases of K, L, M, and N, the pearlite has separated, and the

constituents are ferrite and cementite, the cementite increasing in amount with rise in carbon; micrograph 3. O, shows well laminated pearlite, and free cementite, micrograph 7. R, micrograph 14, shows temper-graphite, ferrite and cementite; and Q, after heating to 1000° , has a similar structure, but with less graphite.

On heating to 1200° and 1300° there is a marked change of structure, particularly in the cases of K, L, M, and N. The constituents are ferrite and pearlite, arranged in a lattice-work structure, with sharp lines of division; micrographs 2 and 5. O still shows pearlite and cementite, but the crystals have greatly increased, and have straight boundaries; micrograph 8. With Q the cementite increases in amount, and occurs plentifully within the pearlite crystals; micrograph 11. In the case of R, after heating to 1300° , all the carbon is combined, and is present as cementite; micrograph 15.

Reheating the overheated bars to 700° does not materially affect their structure.

Reheating to 775° restores the structure nearly to that found before overheating. K, L, M, and N are composed of ferrite and cementite, the cementite increasing in amount with the carbon; micrograph 4. O and Q show pearlite and cementite; micrograph 9; while R, once more has temper-graphite in a matrix of ferrite and cementite; micrograph 16.

CONSIDERATION OF RESULTS.

The effect of the different treatments on the condition of the carbon is full of interest. It would seem as if the rate of cooling had a powerful influence on the separation of the temper-graphite, for Treatment A, and the heating to 1000° , were taken to the same temperature, but cooled at different rates, giving very different results; 0.31 and 1.29 per cent. respectively in the case of R. (Table III.)

Heating to 1000° , followed by very slow cooling, effected the greatest separation of graphite. With increasing temperature less temper-graphite was formed; and after heating to 1300° , all the carbon was combined.

Examining the tables of the mechanical tests, and the dia-

grams, of the steels in which no temper-graphite separated, it is seen that the tests of the steels heated to 1000° and 1100° are very similar. This similarity is also noticed in the microscopical examination.

The marked change in structure seen in K, L, and M after heating to 1200° , is accompanied by an increase in ultimate stress, and a drop in ductility. Heating still further brings about a very coarse crystallization in all cases, an almost complete loss of ductility, and a great lowering in tenacity.

In the microscopical examination it is very noticeable how readily the pearlite of the lower members of the series separates into its constituents. The great change of structure brought about by overheating is therefore more easily recognized.

A change from a cellular to a similar lamellar banded structure was found by R. G. Morse,* when working on a .34 per cent. carbon steel. This great change in structure together with the abrupt change seen in the mechanical tests and fractures on passing from 1100° to 1200° would indicate the presence of some critical point, above which the injurious effects of overheating are very marked. Professor E. Heyn† noticed a similar result, when working on basic open hearth boiler plate steel of .07 per cent. carbon.

The results show that the nickel steels worked on, follow the same laws as ordinary steels with regard to structure and temperature. Among the most important of these are:—(1) The normal crystal size increases with the temperature and (2) The crystal size, even after slow cooling is proportional to the highest temperature reached.

For a full discussion of the laws governing the relation between structure and temperature reference may be made to Professor Howe's recent book.‡

* R. G. Morse. Effect of Heat Treatment upon the Physical Properties and Microstructure of a Medium Carbon Steel. *Transactions American Institute Mining Engineers*, Vol. 29, 1899, p. 729.

† E. Heyn. The Overheating of Mild Steel. *Journal Iron and Steel Institute*, No. II, 1902, p. 73.

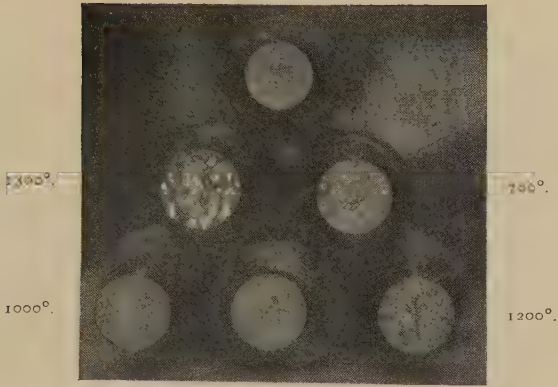
‡ H. M. Howe. Iron, Steel, and Other Alloys.

CONCLUSIONS.

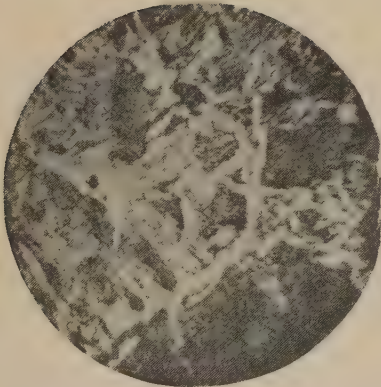
The following conclusions may be drawn from the results:—

1. Steels with a low percentage of nickel follow the same laws regarding structure and temperature as ordinary steels.
2. With the steels up to about 0.80 per cent. carbon, there is a critical point, between 1100° and 1200° , above which the injurious effects of overheating are very marked.
3. In the lower carbon steels overheating brings about a distinct type of structure; ferrite and pearlite arranged in a lattice work, with sharp lines of division.
4. In the higher carbon steels temper-graphite is most liable to be formed by heating to about 1000° followed by slow cooling.
5. With increasing temperature the temper-graphite goes back into combination.

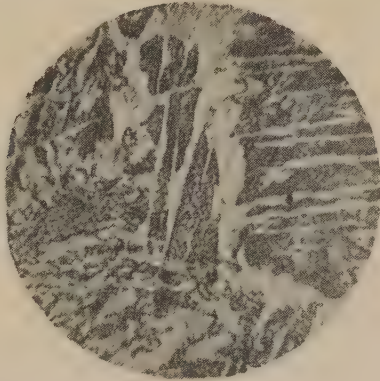
775°.



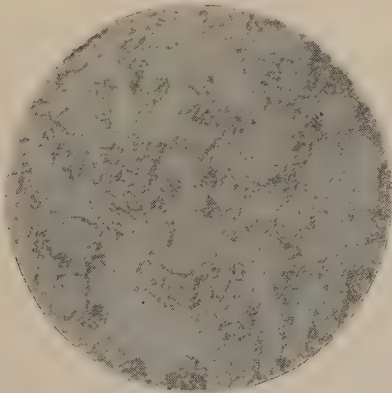
1100°.
Fractures of O.



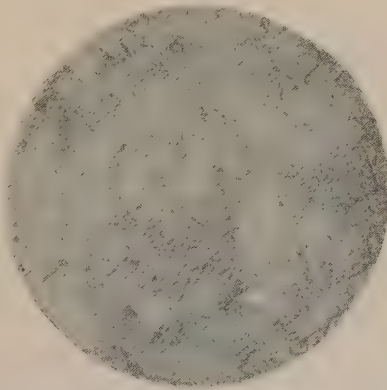
Micrograph 1. K. Treatment A. 225.



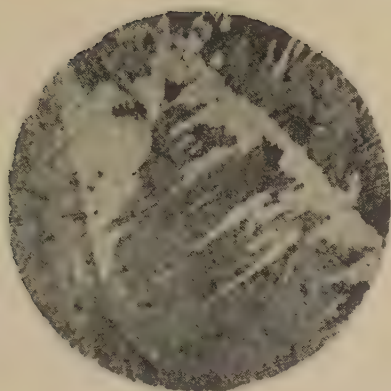
Micrograph 2. K. 1200°. 94.



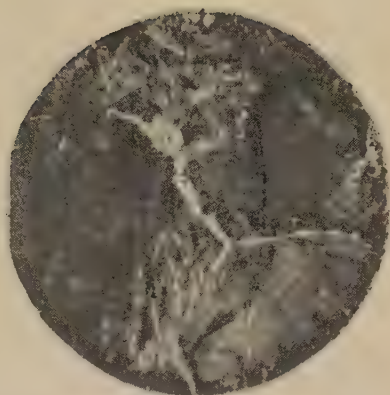
Micrograph 3. L. 1000°. 260.



Micrograph 4. K. 1200° then 775°. 260.



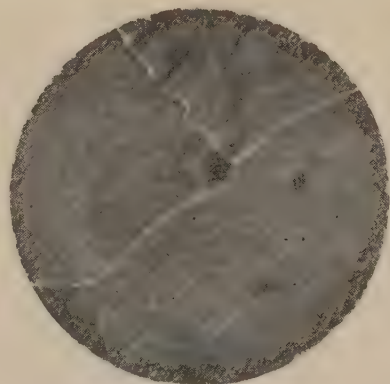
Micrograph 5 L. 1300°. 94.



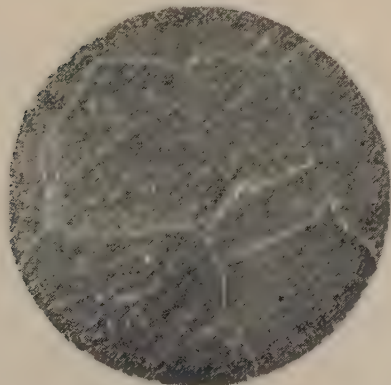
Micrograph 6. M. Treatment A. 225.



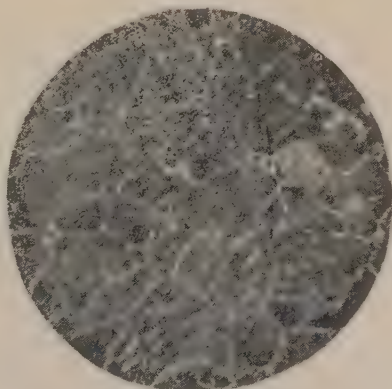
Micrograph 7. O. 1000°. 260.



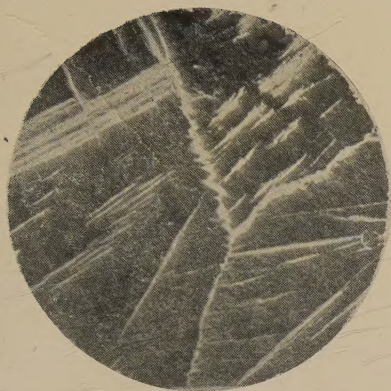
Micrograph 8. O. 1200°. 260



Micrograph 9. O. 1200° then 775°. 260.



Micrograph 10. Q. Treatment A. 260.



Micrograph 11. Q. 1200° 260.



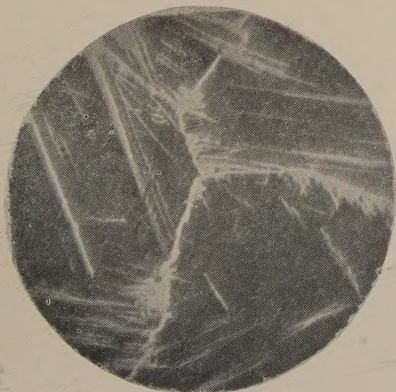
Micrograph 12. R. Treatment A. 260.
Polished.



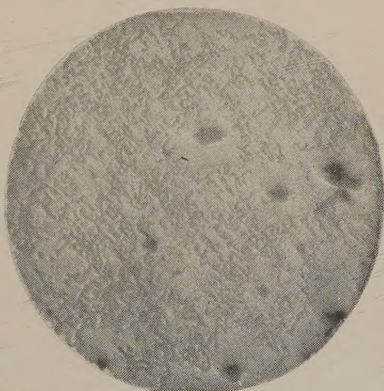
Micrograph 13. R. Treatment A. 340.



Micrograph 14. R. 1000°. 260.



Micrograph 15. R. 1300°. 260.



Micrograph 16. R. 1200° then 775°. 260.

